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Crystal Structure of Cycloheptasarcosyl Hydrate

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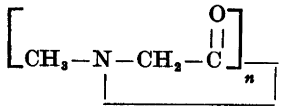
The crystals belong to the monoclinic system with space group $P2_1/c$ and cell dimensions $a=28.725(5)$ Å, $b=11.048(1)$ Å, $c=11.036(1)$ Å, $\beta=132.52(1)°$. The phase problem was solved by direct methods, and the R-value arrived at for 2250 observed reflections was 6.9 % ($R_w=4.9$ %. The conformation is *cis,cis,cis,cis,trans,trans,trans*. Water molecules form hydrogen bond bridges linking molecules to endless chains along [001]. Bond distances and angles are compared with those of cyclopentasarcosyl, cyclooctasarcosyl, and cycloalanyltetrasarcosyl.

Cyclic oligopeptides of sarcosine of the general formula (I) have been studied by Dale and Titlestad.[1-3] To account for the relatively high observed resistance to ring inversion,



$n=2,3,\cdots,8$ (I)

transannular interactions between N and C (carbonyl) were suggested. Recent findings [4-6] do not support this assumption, and the explanation must be sought in the intrinsic conformation of the peptide chain itself.[2] For the case $n=7$ the NMR-spectrum is complex with broad methylene and N-methyl lines which do not become simpler on cooling to −70 °C. Benzene addition resolves seven main N-methyl signals together with several less intense signals, indicating that one major conformation is present. The crystal conformation which is obtained by dissolution in $CHFCl_2$ at −80 °C, shows three N-methyl lines (intensity 9:9:3) and seven partially resolved quartets. In order to settle the conformational problem, and to obtain detailed information of the molecular geometry, and X-ray crystallographic investigation of cycloheptasarcosyl has been carried out.

The crystals belong to the monoclinic system and the systematic absences lead to the space group $P2_1/c$. The cell parameters measured by means of a four circle diffractometer, and their estimated standard deviations are:
$a=28.725(5)$ Å, $b=11.048(1)$ Å, $c=11.036(1)$ Å, $\beta=132.52(1)°$
The unit cell contains four $C_{21}O_7N_7H_{35}.H_2O$ formula units.

With $2\theta_{max}=50°$ and Mo$K\alpha$-radiation 4575 independent reflections were measured on an automatic four-circle diffractometer. Using an observed unobserved cutoff at $2.0\sigma(I)$, 2250 were recorded as observed. No corrections have been made for absorption on secondary extinction effects.

The structure was solved by direct methods [7] and refined by full-matrix least squares technique.[8,*] Hydrogen atom positions were calculated and the positional parameters only were refined. Anisotropic temperature factors were introduced for O, N, and C-atoms, and weights in least squares were calculated from the standard deviations in intensities, $\sigma(I)$, taken as

$$\sigma(I)=[C_T+(0.02C_N)^2]^{\frac{1}{2}}$$

where C_T is the total number of counts and C_N net count (peak minus background). The conventional R-value arrived at was 6.9 % (weighted value $R_w=4.9$ %) for 2250 observed reflections. The form factors were those of Hanson *et al.*[9] except for hydrogen.[10] The final fractional coordinates and thermal vibration parameters are given in Table 1. The expression for anisotropic vibration is:

$$\exp[-(B11h^2+B22k^2+B33l^2+B12hk+B13hl+B23kl)]$$

* All programs used are included in this reference.

Table 1. Final fractional coordinates and anisotropic thermal vibration parameters with estimated standard deviations (multiplied by 10^5 for non-hydrogens and 10^4 for hydrogens). The symbols CC, CM, and OW are used for carbonyl carbons, methyl carbons, and water oxygen, respectively. Hmn is bonded to Cm, HMmn to CMm, and HW to OW.

ATOM	X	Y	Z	B	B11	B22	B33	B12	B13	B23
OW	22986(24)	63964(38)	-5195(62)		343(12)	851(48)	1852(74)	-167(37)	1008(52)	-122(92)
O1	29984(19)	30710(35)	25258(48)		319(12)	672(37)	2310(82)	-207(36)	1156(55)	-810(93)
O2	12689(18)	34963(36)	11709(46)		229(10)	1081(43)	1849(72)	-322(35)	893(49)	247(91)
O3	-1295(17)	46993(37)	-33321(48)		148(10)	1284(47)	2898(79)	-284(36)	449(48)	-662(103)
O4	15893(18)	75289(34)	12803(45)		285(11)	889(40)	1196(64)	-249(35)	662(47)	-198(88)
O5	27396(16)	83998(34)	16178(46)		183(10)	948(42)	1873(74)	43(33)	634(47)	-1174(91)
O6	37588(17)	81549(33)	57259(46)		279(11)	888(40)	2167(77)	150(35)	1286(51)	471(93)
O7	44775(18)	58478(34)	64716(45)		315(12)	790(41)	1634(73)	79(36)	928(51)	579(88)
N1	22578(28)	42332(48)	28744(53)		167(12)	857(48)	1142(76)	-197(39)	510(55)	-133(98)
N2	9208(19)	44288(39)	-15982(50)		164(11)	703(45)	1101(74)	-75(36)	508(52)	-283(93)
N3	7772(19)	71577(37)	-14257(50)		179(11)	644(43)	1079(75)	-84(37)	538(52)	-22(96)
N4	19671(28)	95720(38)	10382(52)		136(10)	503(39)	1286(73)	76(34)	492(49)	-134(90)
N5	36968(19)	96605(38)	42223(48)		143(10)	622(41)	1097(74)	-15(36)	434(49)	144(95)
N6	47087(19)	69497(40)	63186(51)		172(11)	736(45)	1072(75)	65(38)	552(51)	237(96)
N7	36037(20)	46955(40)	31560(50)		186(11)	667(44)	1518(82)	52(39)	745(54)	-286(100)
C1	27483(26)	49186(52)	31316(70)		163(14)	612(52)	1225(93)	15(44)	457(62)	-371(114)
C2	15536(28)	46801(59)	-943(64)		192(14)	855(57)	989(87)	-231(47)	513(63)	252(118)
C3	5071(28)	61740(57)	-12237(78)		177(14)	837(60)	1866(118)	-62(48)	834(78)	-73(134)
C4	14815(26)	88871(51)	-4598(65)		162(14)	564(51)	1281(93)	38(43)	535(65)	176(115)
C5	30598(27)	99819(57)	35208(72)		171(14)	715(54)	1105(89)	-45(45)	468(62)	-514(116)
C6	44997(26)	80747(53)	54048(72)		160(14)	640(53)	1510(101)	-92(45)	684(66)	-192(119)
C7	38158(26)	59179(52)	37659(69)		180(14)	593(51)	1388(95)	-82(44)	673(65)	-39(117)
CC1	31331(25)	41336(50)	29360(61)		173(14)	678(54)	989(86)	1(48)	419(61)	-273(118)
CC2	16842(26)	48621(49)	13971(73)		203(15)	446(48)	1400(99)	-109(44)	715(69)	-171(113)
CC3	4059(25)	60236(58)	-21410(65)		151(14)	766(57)	1447(99)	-115(48)	626(66)	-22(127)
CC4	12519(24)	78058(47)	-1273(65)		156(14)	612(55)	1058(92)	108(45)	415(64)	6(121)
CC5	25714(24)	92291(50)	19720(62)		158(14)	682(56)	1139(91)	-3(45)	517(62)	-146(117)
CC6	39497(23)	86311(46)	51386(62)		134(13)	558(50)	1033(91)	-105(43)	435(59)	-443(109)
CC7	43619(25)	59304(51)	56349(66)		177(14)	715(56)	1364(100)	-15(47)	746(69)	-187(125)
CM1	24378(34)	35385(69)	42366(79)		307(18)	1171(71)	1254(103)	-113(64)	765(77)	93(142)
CM2	8375(36)	32943(66)	-23938(91)		331(19)	902(64)	1969(120)	-42(59)	1315(84)	-593(147)
CM3	4469(33)	74813(64)	-31152(75)		234(16)	944(62)	1199(98)	-149(52)	525(68)	-134(133)
CM4	17640(36)	105198(68)	14958(99)		260(17)	811(60)	2518(136)	318(54)	1394(85)	-342(148)
CM5	38981(31)	101206(68)	33858(83)		199(15)	1008(65)	1497(101)	-94(51)	577(70)	366(134)
CM6	51693(33)	78594(69)	80829(74)		259(17)	1202(71)	1240(101)	-15(58)	618(72)	-355(141)
CM7	39923(37)	39942(69)	30259(90)		365(20)	1126(69)	1964(122)	247(61)	1305(89)	152(151)
HM11	3843(22)	5222(41)	4386(58)	4.0						
HM12	2568(21)	5697(44)	2421(56)	4.0						
HM21	1575(24)	4663(50)	575(61)	4.0						
HM22	1651(22)	5506(44)	-35(57)	4.0						
HM31	95(22)	6324(41)	-1639(56)	4.0						
HM32	730(21)	5989(41)	-91(58)	4.0						
HM41	1668(20)	8606(41)	-929(54)	4.0						
HM42	1130(22)	9502(42)	-1228(55)	4.0						
HM51	3812(21)	10862(44)	3328(55)	4.0						
HM52	3020(22)	9807(42)	4332(57)	4.0						
HM61	4380(21)	7917(42)	4348(58)	4.0						
HM62	4849(22)	8665(42)	5974(55)	4.0						
HM71	3481(22)	6430(42)	3547(56)	4.0						
HM72	3976(21)	6334(42)	3254(54)	4.0						
HM11	2128(22)	3115(44)	4887(55)	4.0						
HM12	2655(22)	4814(42)	5235(59)	4.0						
HM13	2783(22)	2906(44)	4693(56)	4.0						
HM21	487(22)	3291(42)	-3592(59)	4.0						
HM22	914(21)	2558(44)	-1690(57)	4.0						
HM23	1135(21)	3166(44)	-2457(56)	4.0						
HM31	685(21)	8012(45)	-3288(54)	4.0						
HM32	4(23)	7615(42)	-3800(56)	4.0						
HM33	449(22)	6802(45)	-3656(57)	4.0						
HM41	2047(22)	11028(44)	2513(58)	4.0						
HM42	1520(24)	10443(48)	1507(65)	4.0						
HM43	1605(23)	11120(45)	888(58)	4.0						
HM51	4354(23)	10243(43)	4152(57)	4.0						
HM52	3741(21)	10973(46)	3029(56)	4.0						
HM53	3686(22)	9728(44)	2365(59)	4.0						
HM61	5299(22)	6332(44)	8517(56)	4.0						
HM62	5008(21)	7380(44)	8530(56)	4.0						
HM63	5523(23)	7586(43)	8459(55)	4.0						
HM71	3768(22)	3305(45)	2371(56)	4.0						
HM72	4398(23)	3817(42)	4095(58)	4.0						
HM73	4059(22)	4371(44)	2440(58)	4.0						
HW1	2048(22)	6725(47)	-1454(58)	4.0						
HW2	2438(23)	7004(44)	-126(62)	4.0						

The principal axes of the thermal vibration ellipsoids for oxygen, nitrogen, and carbon atoms were calculated from the temperature parameters of Table 1. Maximum root mean squares amplitudes range from about 0.22 Å for ring atoms to about 0.33 Å for methyl carbon atoms and the water oxygen. Due to the size of the molecule, no rigid-body analysis of translational, librational, and screw motion has been carried out. A comparison between observed and calculated structure factors is presented in Table 2.

Interatomic distances, bond angles and dihedral angles are given in Table 3. The standard deviations, given in parentheses, are estimated from the correlation matrix of the last least

Table 2. Observed and calculated structure factors on 10 times absolute scale.

K=	0,L=12		10	1151	1207	11	84	7	11	550	535	13	210	203	7	79	54	6	291	287	16	90	76
11	183	163	11	428	435	12	91	132	12	262	282	14	152	149	9	142	157	8	544	524	17	160	145
12	96	57	12	343	345	13	208	195	14	242	231	15	390	384	12	121	108	9	277	265	19	112	122
19	138	157	13	74	47	23	181	193	15	400	388	18	116	126	18	89	100	10	351	362	20	118	107
20	90	88	14	622	619	25	223	230	16	371	373	19	145	163	20	166	183	12	365	357	K=	2,L=	1
24	93	93	15	232	232	26	147	148	18	292	289	20	103	121	21	147	167	13	256	248	0	1343	1328
26	92	104	16	361	352	28	141	142	19	219	221	K=	1,L=	2	23	177	186	14	127	126	1	530	535
28	89	8	17	218	240	29	88	67	23	96	117	0	440	416	24	212	210	15	569	558	3	194	210
29	93	54	18	79	95	30	95	67	24	129	119	1	310	326	25	164	157	16	638	635	4	489	511
K=	0,L=10		21	113	84	32	90	45	25	161	150	2	536	515	26	110	87	17	230	221	5	712	706
7	115	97	22	255	248	33	110	79	26	86	104	3	614	637	27	127	127	18	184	140	6	967	957
8	132	130	23	367	350	34	138	75	27	134	127	5	109	139	29	106	94	19	244	248	7	411	393
11	181	190	K=	0,L=	0	K=	1,L=	8	30	114	77	6	284	268	30	100	60	20	195	206	8	220	202
13	171	155	2	359	368	1	81	105	K=	1,L=	3	7	104	90	32	124	104	21	116	117	10	193	170
15	250	244	3	193	215	2	103	73	1	1000	994	8	146	181	K=	2,L=	8	23	122	145	11	237	218
18	133	152	4	564	575	7	83	107	3	371	352	9	145	168	1	83	47	26	356	356	12	84	58
20	134	125	5	261	267	9	85	131	4	706	665	11	331	334	4	92	93	27	166	141	13	108	98
24	96	81	6	341	350	10	107	94	5	519	507	12	272	262	5	136	131	28	135	128	14	143	150
25	168	183	7	365	361	11	86	87	7	85	84	16	98	113	6	131	97	K=	2,L=	3	15	179	177
26	113	108	8	447	452	12	121	135	8	711	725	19	113	101	7	80	64	1	906	895	16	131	118
27	193	215	9	717	750	13	382	374	9	583	580	K=	1,L=	3	8	182	189	2	88	108	17	192	193
29	189	180	10	308	323	14	165	159	10	410	409	0	856	864	11	143	147	3	491	506	18	108	108
31	198	208	11	291	304	16	208	216	11	511	497	2	690	701	13	209	219	4	288	263	20	88	64
K=	0,L=	8	12	558	565	19	125	132	13	407	395	3	105	78	14	194	191	5	300	319	K=	2,L=	2
1	159	167	13	743	739	21	83	85	15	140	81	4	210	203	15	186	195	6	760	724	0	493	506
2	81	107	15	210	208	25	119	141	16	108	120	5	606	593	16	120	104	7	822	800	1	819	782
3	299	294	16	141	130	26	204	205	17	111	68	6	148	145	17	100	97	8	289	298	2	492	492
4	369	370	17	226	241	33	117	111	18	193	191	7	304	305	18	266	269	9	913	909	3	217	223
6	181	173	22	108	112	K=	1,L=	7	20	142	148	9	160	163	19	190	186	10	869	870	4	487	475
7	345	334	23	152	145	1	84	71	21	169	177	11	305	281	21	159	171	11	399	408	5	514	519
8	151	166	25	149	138	2	267	272	23	421	411	13	102	96	22	173	172	12	357	347	6	240	244
10	121	119	K=	0,L=	2	4	77	73	24	146	136	15	119	120	25	157	141	13	139	112	7	110	107
11	193	215	0	836	851	5	152	150	25	89	111	K=	1,L=	4	26	85	17	14	131	85	9	283	276
13	151	119	2	600	614	6	74	69	K=	1,L=	2	0	188	193	29	145	179	16	307	311	11	327	328
16	258	250	3	926	929	7	314	318	1	1562	1554	1	569	591	K=	2,L=	7	17	134	117	14	215	221
17	266	261	4	647	659	8	106	119	2	132	135	2	395	411	1	347	342	19	95	70	16	89	95
20	108	120	5	537	551	10	76	103	3	460	486	3	128	124	3	193	200	20	157	157	18	176	165
21	227	242	6	959	949	11	278	244	4	730	751	4	231	228	4	177	186	24	124	127	20	90	73
22	403	420	7	488	483	12	484	491	5	357	347	7	221	222	5	124	103	25	188	206	K=	2,L=	3
23	269	294	9	88	98	13	100	106	7	500	488	10	250	255	6	189	178	27	91	71	0	84	114
24	117	117	10	110	131	14	201	210	8	408	410	11	136	114	8	234	237	28	110	102	2	273	258
26	101	89	12	256	246	15	229	222	10	307	298	12	117	133	9	266	265	29	104	98	3	303	306
27	246	250	13	140	147	17	90	77	11	172	151	14	106	56	11	127	124	K=	2,L=	2	4	276	289
28	102	87	14	414	430	19	118	119	12	301	279	15	125	108	12	403	402	1	805	586	5	216	238
31	89	86	15	129	146	20	389	375	13	250	239	K=	1,L=	5	14	263	284	2	802	841	7	114	113
K=	0,L=	6	K=	0,L=	4	21	82	79	14	608	588	0	155	177	19	119	102	3	1001	1012	10	86	105
1	328	334	0	850	848	23	379	398	16	173	164	1	124	112	20	296	292	4	869	863	11	115	113
2	111	116	1	149	175	24	376	394	17	290	298	2	217	213	21	227	231	5	128	156	13	214	246
3	186	193	2	82	87	25	146	136	18	139	138	3	271	274	28	162	198	6	298	301	17	109	96
4	379	390	3	228	221	27	170	167	19	177	162	6	174	206	29	184	186	7	82	97	K=	2,L=	4
5	121	108	4	87	123	29	123	117	20	233	231	7	78	43	33	90	98	8	1056	1058	0	186	197
6	307	305	6	293	302	K=	1,L=	6	21	94	91	8	126	113	K=	2,L=	8	9	1387	1402	1	71	70
7	147	146	7	105	72	1	98	132	22	133	127	9	97	80	1	116	130	10	366	346	3	138	109
9	696	716	8	97	99	2	120	107	23	141	134	11	86	47	3	77	67	11	865	857	4	176	174
10	346	351	9	379	396	3	372	362	K=	1,L=	1	12	97	105	5	214	200	12	585	594	5	267	271
11	235	251	11	430	424	4	443	437	1	182	191	K=	1,L=	6	6	297	273	13	205	197	6	83	33
12	649	685	14	128	146	5	178	180	2	349	338	0	855	864	8	126	129	15	253	261	7	92	118
13	80	51	16	150	142	6	133	140	3	256	262	1	144	167	9	109	102	18	120	135	9	161	145
14	225	235	K=	0,L=	6	8	390	371	4	831	837	2	115	128	10	260	264	19	89	101	10	306	311
15	105	92	0	400	400	10	97	92	5	1018	1026	3	181	200	11	277	273	21	182	194	11	260	273
16	353	344	1	485	490	11	96	85	7	849	842	5	141	149	13	605	617	22	314	301	12	128	105
17	117	168	2	401	398	15	146	135	8	393	374	6	218	221	14	106	118	23	207	210	14	141	136
22	116	135	3	149	141	16	237	224	10	466	456	7	96	104	15	208	204	24	109	114	16	110	94
25	330	318	4	220	211	18	138	136	11	124	125	K=	1,L=	7	17	102	104	K=	2,L=	1	K=	2,L=	5
26	171	165	5	275	288	21	195	235	12	313	326	0	163	179	18	99	93	1	344	325	0	133	127
27	138	159	7	199	229	22	295	301	13	429	438	2	156	163	20	205	198	2	1542	1500	1	152	177
K=	0,L=	4	K=	0,L=	8	23	221	229	16	153	134	K=	1,L=	9	22	84	116	3	311	325	4	245	240
2	923	922	1	110	124	24	164	182	17	115	155	1	104	92	23	89	84	4	1076	1064	5	222	223
3	1100	1118	2	135	192	26	166	169	18	281	295	K=	2,L=	13	24	204	206	5	412	420	6	112	107
4	562	566	3	134	118	K=	1,L=	5	19	302	309	23	189	181	25	219	236	6	928	933	7	104	100
5	456	453	5	115	108	1	298	293	20	193	190	K=	2,L=	12	27	142	153	7	107	117	9	266	248
7	102	87	K=	1,L=	12	2	255	231	22	97	69	13	118	83	28	83	21	8	355	357	11	133	130
9	574	570	19	159	154	3	413	399	23	116	142	14	83	24	30	100	119	9	108	123	K=	2,L=	6
10	307	308	22	116	108	5	385	373	K=	1,L=	0	16	87	57	K=	2,L=	5	10	183	194	0	174	180
11	71	62	28	106	127	6	357	376	1	588	572	18	84	52	2	338	332	11	423	438	2	90	122
12	414	429	K=	1,L=	11	7	96	93	3	921	921	23	98	121	3	367	358	12	535	526	3	144	140
13	115	72	14	146	146	8	467	445	4	347	341	K=	2,L=	11	5	88	49	13	83	86	5	150	116
14	471	474	17	131	111	9	494	488	5	509	513	18	104	109	6	212	198	14	258	263	6	169	165
15	143	160	19	115	126	12	218	206	6	447	466	22	107	117	7	174	158	15	141	139	7	196	206
16	134	139	22	208	203	13	100	112	8	330	311	26	101	40	9	177	169	16	127	110	8	82	14
17	114	166	23	108	99	14	166	155	9	714	693	27	123	106	10	600	576	17	102	101	K=	2,L=	7
19	135	153	25	127	120	15	234	264	10	330	322	K=	2,L=	10	11	78	62	18	128	112	0	91	63
20	180	162	K=	1,L=	10	17	482	479	11	170	174	2	102	102	12	88	68	21	140	127	1	85	88
21	130	163	8	140	114	19	472	490	13	337	332	7	141	146	13	179	161	23	149	137	2	106	81
22	102	97	15	146	164	20	170	145	16	315	304	10	163	160	14	82	96	24	112	111	5	82	41
23	88	129	16	145	144	21	375	370	17	135	116	11	124	134	15	620	644	25	131	109	K=	2,L=	8
26	202	191	19	129	138	22	353	366	18	118	113	12	174	177	16	271	285	K=	2,L=	0	0	105	127
27	218	216	20	84	107	24	148	159	22	117	96	14	87	84	18	278	284	0	962	943	3	88	89
29	92	68	21	82	75	26	83	33	K=	1,L=	1	15	92	49	22	239	234	1	249	259	K=	2,L=	9
30	92	15	24	292	280	27	86	38	1	248	248	16	107	90	23	104	110	2	708	739	0	120	121
31	132	116	27	87	85	K=	1,L=	4	2	650	671	18	102	67	24	130	99	3	745	745	K=	3,L=	12
K=	0,L=	2	28	99	97	1	139	114	3	604	608	19	164	152	25	287	290	4	157	199	22	90	23
1	529	560	K=	1,L=	9	2	337	321	4	651	639	21	97	131	26	117	83	5	1219	1212	K=	3,L=	11
2	550	583	2	88	106	3	103	114	5	453	444	23	102	92	31	96	74	6	285	281	9	100	84
3	1450	1451	3	82	9	4	432	426	6	72	78	25	186	199	K=	2,L=	4	7	132	149	10	134	114
5	829	816	5	92	78	5	503	494	7	130	129	26	140	144	1	610	600	8	135	133	14	161	152
6	1394	1389	6	152	143	6	307	310	9	565	541	27	143	121	2	144	155	10	171	139	15	127	120
7	1644	1747	7	170	164	7	274	263	10	219	205	29	105	102	3	429	420	11	442	425	19	125	144
8	739	770	8	93	81	9	220	214	11	352	358	31	95	45	4	451	452	14	88	91	22	118	128
9	1021	1035	10	232	246	10	608	608	12	135	116	K=	2,L=	9	5	388	385	15	299	284	24	159	156

Table 2. Continued.

26	132	137	26	154	124	6	101	75	9	373	363	24	169	158	7	100	89	8	316	310	9	239	226
28	93	91	28	171	187	7	394	410	11	247	251	25	124	141	8	80	14	9	125	130	12	253	238
K=	3,L=	10	30	112	100	8	160	164	12	281	291	27	107	99	9	116	109	10	270	271	13	257	272
8	89	72	K=	3,L=	4	9	355	363	13	95	103	28	107	100	12	140	112	11	269	269	14	210	204
10	286	281	1	180	168	11	113	110	14	142	126	K=	4,L=	2	13	172	196	12	100	79	15	239	231
12	151	121	3	290	267	12	126	149	16	115	114	1	92	71	K=	4,L=	5	14	89	51	16	148	135
13	185	160	4	167	185	14	125	138	17	135	149	3	185	181	0	437	440	15	267	260	17	288	278
20	93	67	5	388	380	15	405	424	18	164	178	4	989	981	1	213	213	16	141	137	K=	5,L=	2
21	91	74	6	655	652	16	202	200	19	113	135	6	373	381	2	313	325	17	113	82	0	338	323
22	113	77	7	300	287	18	155	151	23	100	132	7	440	440	6	203	214	18	93	56	1	362	371
23	99	67	8	608	618	20	132	131	24	185	170	8	180	194	7	82	68	19	206	202	2	220	187
24	85	12	9	109	122	21	147	137	26	86	61	9	301	287	9	103	124	20	167	197	3	448	428
25	96	102	10	390	377	22	128	115	27	96	105	10	421	447	K=	4,L=	6	22	191	196	4	152	170
28	89	45	11	424	395	K=	3,L=	2	32	123	112	11	203	211	0	285	299	23	91	93	5	176	181
32	107	47	12	179	170	0	312	310	K=	4,L=	7	12	367	365	1	86	62	27	145	118	7	88	85
K=	3,L=	9	13	816	803	2	261	249	1	209	210	17	196	193	10	93	96	29	120	118	8	253	261
4	132	92	14	296	304	3	301	287	4	177	180	18	149	171	K=	4,L=	7	K=	5,L=	4	9	122	103
5	102	118	15	200	210	4	238	206	6	190	202	19	134	123	1	108	90	1	368	372	11	150	146
6	241	233	16	160	153	5	340	324	7	177	172	20	180	184	3	105	99	2	82	79	13	138	145
8	119	145	17	135	130	6	179	181	8	96	106	22	141	151	5	90	72	3	266	279	16	126	125
9	137	139	21	145	137	7	249	245	9	307	306	K=	4,L=	1	K=	4,L=	8	5	174	174	K=	5,L=	3
10	244	258	23	162	165	8	225	215	10	131	145	1	585	561	2	97	92	6	279	257	0	88	78
12	114	94	30	92	20	9	90	102	11	121	96	2	243	236	3	148	145	7	310	301	1	72	55
13	211	211	K=	3,L=	3	10	307	302	12	78	96	3	83	67	K=	4,L=	9	8	137	115	2	99	86
15	92	95	2	272	258	13	156	176	14	320	333	4	96	42	0	121	127	9	208	244	3	331	327
16	87	82	3	866	859	14	147	151	15	216	214	5	360	342	K=	5,L=	12	10	448	468	5	114	110
17	189	202	4	248	246	15	104	87	16	157	156	6	551	537	18	128	107	12	120	146	6	77	8
18	115	102	5	537	521	17	159	161	17	247	244	7	296	274	K=	5,L=	11	13	220	200	7	261	275
19	90	85	6	498	504	18	158	149	21	194	197	9	424	420	14	140	147	14	150	188	9	97	87
20	91	120	7	728	706	19	103	100	24	152	152	10	273	267	17	122	106	15	256	271	10	438	452
21	147	163	8	269	272	K=	3,L=	3	26	116	100	12	132	92	22	155	131	17	188	169	11	304	314
22	189	209	10	135	113	0	261	265	32	105	17	13	150	140	25	95	47	18	172	185	14	134	95
23	260	242	11	333	341	1	835	840	K=	4,L=	6	14	422	439	K=	5,L=	10	19	191	175	K=	5,L=	4
27	146	137	12	166	155	2	94	93	1	119	117	15	100	51	7	88	66	20	95	73	0	233	224
28	87	92	13	91	74	4	312	313	2	188	179	16	125	115	14	136	144	21	181	170	2	86	90
29	86	81	14	334	343	5	301	297	3	221	229	18	116	122	21	155	105	24	215	208	3	230	228
K=	3,L=	8	15	178	198	6	336	343	4	106	102	19	98	103	23	137	127	K=	5,L=	3	4	359	379
2	294	299	16	80	87	7	119	133	6	79	69	20	137	129	24	100	3	1	192	159	5	179	174
3	109	104	17	182	162	9	176	184	8	122	138	21	338	346	K=	5,L=	9	2	246	226	7	127	125
4	333	335	20	92	103	10	249	251	12	293	287	24	94	28	4	134	124	3	100	130	8	91	119
5	203	191	23	150	124	12	210	194	13	406	404	K=	4,L=	0	6	84	58	4	237	219	11	81	11
6	364	372	24	172	187	13	146	170	14	169	151	0	362	360	9	89	83	5	292	283	12	180	172
9	114	118	26	107	80	14	253	256	18	257	272	1	150	145	10	206	207	6	651	641	K=	5,L=	5
11	135	114	K=	3,L=	2	15	96	57	21	119	146	2	631	656	11	86	73	7	288	278	0	108	105
14	118	122	1	899	939	16	150	148	22	121	116	3	221	238	14	90	79	8	209	198	1	148	159
15	227	220	3	93	81	17	116	91	K=	4,L=	5	5	231	244	15	129	120	9	101	136	2	274	274
18	85	87	4	535	534	K=	3,L=	4	1	192	179	6	640	650	16	179	174	10	276	276	3	244	254
23	103	112	5	434	438	0	422	430	2	254	261	7	123	122	18	137	128	11	454	458	4	272	286
27	126	109	6	1578	1615	1	324	345	3	135	131	8	292	288	22	121	133	13	200	193	5	122	101
K=	3,L=	7	7	395	414	2	502	507	4	224	246	9	82	42	23	97	111	14	198	183	6	188	191
2	151	170	8	230	223	6	97	60	7	287	264	10	103	74	K=	5,L=	8	15	262	258	11	144	141
3	292	294	9	910	921	7	190	191	9	180	179	11	358	354	4	92	83	16	194	188	K=	5,L=	6
5	205	205	10	427	408	8	108	121	10	128	121	12	219	221	8	198	200	17	153	154	1	259	269
6	121	139	11	182	137	10	147	140	11	646	664	14	149	152	10	112	123	18	113	103	2	84	44
7	104	115	12	546	540	11	111	117	12	95	93	15	97	100	13	276	279	19	256	261	4	105	136
8	209	211	13	187	200	15	125	110	13	108	73	16	178	166	15	342	346	23	107	82	K=	5,L=	7
10	256	276	16	104	109	K=	3,L=	5	14	85	75	19	92	71	16	119	124	K=	5,L=	2	0	264	264
11	158	150	18	143	137	1	379	390	15	132	137	K=	4,L=	1	17	174	180	1	317	329	5	89	95
12	188	177	19	168	181	2	145	140	17	219	213	0	206	201	18	115	136	3	232	218	K=	5,L=	8
13	110	104	20	140	140	3	127	118	19	118	97	1	141	145	20	100	102	4	285	281	2	94	101
15	119	118	23	111	106	9	169	160	20	408	404	2	319	307	21	270	259	5	140	137	K=	6,L=	11
16	287	277	24	119	96	10	96	95	22	89	102	3	287	290	23	93	63	8	176	131	16	109	120
17	182	191	25	208	206	13	117	83	23	102	125	4	552	550	K=	5,L=	7	9	343	352	K=	6,L=	10
19	344	343	K=	3,L=	1	K=	3,L=	6	25	100	104	5	709	692	1	95	96	10	145	145	11	135	96
24	150	149	1	353	349	0	329	317	28	120	115	6	134	121	2	134	153	11	461	458	13	150	132
25	129	122	3	337	332	1	334	334	K=	4,L=	4	7	332	325	3	162	155	12	389	386	14	111	76
26	194	187	4	396	377	2	196	210	1	517	491	10	291	273	4	99	100	14	199	206	15	100	107
28	131	140	6	319	319	3	196	205	2	175	172	11	201	213	7	126	102	17	199	166	20	85	66
31	120	98	8	108	84	5	112	97	3	431	424	14	241	256	9	229	223	18	128	117	24	90	70
K=	3,L=	6	9	215	211	K=	3,L=	7	4	220	222	15	122	125	10	84	102	20	170	147	26	89	14
1	116	102	10	304	298	1	143	154	6	211	201	16	232	237	12	365	375	21	187	208	K=	6,L=	9
3	83	76	11	443	440	2	105	78	7	166	160	17	106	95	14	138	155	22	80	42	6	90	73
4	89	49	12	516	506	3	140	140	8	294	299	22	93	59	15	120	135	K=	5,L=	1	8	122	130
6	135	126	13	117	97	K=	4,L=	11	10	339	348	K=	4,L=	2	17	126	129	1	594	585	10	165	178
7	263	257	14	225	225	12	95	86	11	214	248	0	539	506	18	103	129	2	106	88	13	258	255
8	164	178	15	449	429	13	90	68	12	162	170	1	83	73	20	100	69	3	324	316	15	86	80
9	264	237	18	92	75	20	106	92	13	510	480	2	425	427	21	116	124	4	123	134	22	140	151
10	286	269	19	372	361	21	102	95	15	221	216	3	127	144	22	90	83	5	127	137	K=	6,L=	8
11	260	251	20	159	164	23	123	112	16	158	149	4	294	308	23	122	137	6	219	208	2	112	107
12	291	99	22	97	68	24	164	157	17	217	235	5	275	282	24	116	115	8	131	123	3	98	88
14	90	106	23	109	103	K=	4,L=	10	18	203	198	7	196	199	26	48	143	11	80	24	6	106	116
15	90	67	26	112	126	10	215	203	20	130	161	8	98	108	K=	5,L=	6	12	355	339	8	179	166
16	110	90	K=	3,L=	0	13	230	250	22	168	158	11	221	241	1	192	197	13	179	174	9	202	221
17	203	202	1	589	592	14	209	208	23	104	100	12	284	273	2	176	173	15	90	42	11	231	244
20	150	137	2	856	854	16	85	59	24	167	181	16	100	91	3	120	135	17	123	121	12	213	194
21	189	186	3	460	469	18	138	138	29	97	85	17	110	130	4	212	212	20	102	97	14	140	140
22	127	144	5	472	473	19	129	140	K=	4,L=	3	19	149	138	5	110	107	21	136	142	15	85	62
23	196	194	6	228	224	21	154	163	1	620	611	K=	4,L=	3	6	81	94	K=	5,L=	0	16	128	122
29	108	160	7	139	138	22	210	199	2	458	459	0	74	65	7	284	297	1	155	160	18	162	195
K=	3,L=	5	8	139	110	K=	4,L=	9	3	429	432	2	296	265	9	101	107	2	447	453	19	179	175
1	233	229	9	96	62	2	103	94	4	280	263	4	324	322	10	395	389	3	228	222	23	153	148
2	121	154	11	199	214	8	162	156	5	207	203	5	170	170	12	278	288	4	169	160	24	133	144
3	147	145	12	235	239	9	111	70	6	616	623	7	241	238	13	138	124	6	146	131	29	94	70
4	524	535	13	104	126	10	134	125	7	227	205	8	112	98	14	86	66	9	145	176	K=	6,L=	7
7	85	109	14	175	151	13	115	144	8	306	307	9	82	69	15	168	157	10	215	242	2	117	113
8	516	523	16	137	139	14	164	159	9	620	634	11	142	129	17	107	75	12	116	115	3	149	137
9	516	518	17	174	190	18	194	204	10	209	198	12	258	255	20	192	193	13	125	129	9	290	266
10	445	433	18	128	109	19	95	89	11	365	346	13	147	159	23	116	102	14	134	127	10	160	184
11	564	565	21	211	225	21	148	143	12	172	154	16	94	10	24	146	165	15	138	141	11	211	218
12	434	424	22	131	131	24	131	127	13	79	65	17	88	94	26	152	141	16	139	120	16	92	58
17	257	235	K=	3,L=	1	25	151	153	14	239	237	K=	4,L=	4	K=	5,L=	8	17	352	363	18	191	226
18	187	205	0	426	399	27	87	73	15	289	301	0	222	225	1	280	290	23	112	143	19	118	150
20	138	126	1	480	475	K=	4,L=	8	16	525	529	1	86	67	2	212	216	K=	5,L=	1	22	127	84
21	84	140	2	711	704	1	84	70	17	94	123	2	186	188	4	79	73	0	308	314	25	109	27
22	256	256	3	427	425	3	147	146	20	108	96	3	287	294	5	83	108	3	560	559	K=	6,L=	6
23	81	63	4	498	507	7	229	255	21	156	171	4	135	124	6	245	233	4	578	567	1	120	132
24	244	253	5	775	775	8	95	109	23	298	298	5	145	153	7	84	54	5	177	169	5	273	275

Table 2. Continued.

7	311	331	3	400	403	16	154	158	1	102	125	11	135	166	2	102	76	10	229	230	1	212	203
9	192	181	4	245	232	18	210	220	2	377	410	12	145	113	3	101	79	13	161	151	2	189	210
11	86	58	5	205	193	19	90	100	3	91	106	13	83	24	6	104	69	K=	9,L=	2	3	133	128
12	312	308	7	90	90	21	112	130	4	123	130	14	373	379	K=	8,L=	6	2	89	121	5	99	95
14	291	308	8	119	151	22	135	153	5	168	187	15	109	138	3	111	101	3	106	115	8	205	201
15	258	273	10	88	60	23	152	145	7	226	223	16	126	88	K=	9,L=	9	4	92	100	10	123	78
16	90	100	11	206	222	24	105	109	8	205	211	21	115	145	11	87	28	6	166	176	11	143	140
17	139	126	12	369	389	27	95	89	10	240	248	22	92	79	19	109	78	7	196	196	K=	10,L=	3
18	239	253	15	89	72	K=	7,L=	5	11	133	158	K=	8,L=	3	21	94	47	8	88	63	0	123	134
20	148	175	K=	6,L=	1	2	322	318	12	335	353	1	140	133	K=	9,L=	8	9	136	140	3	133	126
24	305	309	0	149	126	4	105	104	13	96	75	2	167	163	12	137	117	10	102	101	4	103	106
K=	6,L=	5	1	348	357	6	144	142	K=	7,L=	2	3	79	96	13	112	90	K=	9,L=	3	5	101	8
1	113	113	2	154	126	8	251	265	0	181	201	4	131	132	21	137	136	0	179	190	6	141	138
3	130	128	3	127	123	11	250	268	1	95	93	5	190	177	22	92	74	1	104	131	7	97	89
4	138	148	6	158	156	12	166	136	2	195	202	6	108	86	K=	9,L=	7	3	128	113	10	100	88
5	137	167	7	205	226	13	86	97	4	132	156	7	273	266	6	123	98	5	96	65	K=	10,L=	4
6	290	281	8	108	75	14	174	157	7	186	208	8	176	171	9	100	70	6	185	173	8	116	126
7	467	486	9	116	113	15	204	200	8	175	178	9	97	108	10	94	62	7	99	75	K=	10,L=	5
8	164	178	10	139	158	20	100	105	9	144	142	10	347	347	11	84	13	12	95	21	3	100	74
9	288	293	11	140	151	21	87	118	10	103	138	12	180	185	13	96	68	K=	9,L=	4	K=	10,L=	6
10	284	286	12	305	306	26	109	68	11	218	223	14	86	83	21	103	83	0	86	99	0	92	26
12	147	152	13	185	186	K=	7,L=	4	16	107	88	16	299	307	23	94	65	1	116	131	K=	11,L=	7
13	192	190	18	111	100	1	239	249	K=	7,L=	3	20	91	80	K=	9,L=	6	2	255	248	11	115	100
14	146	160	K=	6,L=	2	2	78	36	3	163	155	K=	8,L=	2	2	144	154	4	153	162	K=	11,L=	6
15	80	44	3	340	354	3	137	142	4	91	86	1	132	121	5	174	182	K=	9,L=	6	10	122	69
16	165	148	5	112	127	4	80	66	5	121	111	2	97	72	13	261	245	0	146	137	K=	11,L=	9
18	96	80	6	99	103	5	168	183	6	96	108	5	223	234	15	199	166	K=	10,L=	8	9	147	144
19	140	135	8	294	299	7	146	174	8	94	78	6	195	226	16	128	102	15	137	101	11	112	93
20	102	93	11	264	257	8	99	80	10	86	60	7	95	88	17	190	224	K=	10,L=	7	12	172	153
22	138	144	13	204	198	9	141	153	11	117	99	8	81	39	18	106	104	6	94	60	13	146	124
23	130	136	K=	6,L=	3	13	181	206	12	122	141	10	317	320	K=	9,L=	5	9	92	31	18	96	26
25	140	154	0	173	176	14	134	144	13	93	82	11	219	248	1	90	78	12	99	119	K=	11,L=	4
26	93	73	1	198	227	15	185	185	14	133	100	12	262	243	3	152	167	K=	10,L=	6	4	187	179
K=	6,L=	4	2	142	163	20	193	210	K=	7,L=	4	13	106	132	12	237	236	3	99	69	16	108	71
1	176	196	3	143	158	21	90	93	0	83	93	14	146	160	13	84	86	17	120	120	K=	11,L=	3
2	113	119	4	97	120	23	123	140	1	93	29	16	113	113	19	96	75	K=	10,L=	5	2	141	130
3	101	99	5	137	147	K=	7,L=	3	4	96	103	18	84	73	20	86	24	5	124	103	5	187	218
5	108	99	6	117	161	1	153	140	5	83	96	K=	8,L=	1	K=	9,L=	4	6	91	59	9	175	175
8	255	266	7	222	225	2	248	247	6	121	134	2	365	368	1	113	106	7	153	124	10	178	162
8	141	170	8	92	85	5	115	115	11	144	174	3	97	106	3	84	75	8	113	119	16	112	82
9	77	52	9	105	97	6	284	293	K=	7,L=	5	4	220	228	5	143	111	10	119	124	K=	11,L=	2
10	220	197	K=	6,L=	4	7	117	130	0	93	103	6	104	95	7	252	253	12	146	158	1	144	264
11	308	297	1	207	208	9	114	117	1	112	107	7	139	159	9	99	100	14	118	134	4	86	36
12	304	311	3	160	165	10	140	134	3	111	98	8	162	189	10	146	140	K=	10,L=	4	8	101	83
13	118	121	4	293	260	11	321	333	K=	7,L=	6	9	173	195	11	392	388	1	245	241	12	127	115
14	123	108	5	151	140	14	154	176	3	150	132	10	130	127	12	360	373	2	233	243	K=	11,L=	1
15	345	337	8	224	229	15	100	80	5	89	59	11	125	113	14	129	132	3	82	64	1	207	213
16	110	90	9	283	265	16	240	227	6	117	82	12	318	340	15	236	254	5	100	56	5	100	97
20	113	128	10	85	66	18	135	146	7	112	115	13	223	237	18	172	166	7	84	77	6	200	187
21	137	144	13	92	21	19	187	190	K=	7,L=	7	14	172	183	20	136	134	8	103	138	10	102	77
23	132	119	K=	6,L=	5	20	178	171	4	123	125	16	125	119	23	98	94	13	166	212	K=	11,L=	0
24	144	103	1	217	221	23	101	81	K=	7,L=	8	K=	8,L=	0	K=	9,L=	3	14	152	159	2	105	114
K=	6,L=	3	3	250	255	K=	7,L=	2	0	100	100	1	157	140	1	120	130	15	121	92	3	141	172
1	210	188	4	82	57	1	225	222	K=	8,L=	9	2	116	109	2	167	152	18	94	105	8	172	166
4	224	217	11	103	22	2	125	106	7	96	30	3	115	88	4	94	109	K=	10,L=	3	K=	11,L=	1
5	493	476	K=	6,L=	6	3	84	129	14	102	66	5	144	134	5	149	145	1	170	155	2	124	99
7	94	101	1	163	188	4	253	252	16	151	154	7	196	219	7	109	90	2	231	222	9	105	100
8	166	157	3	122	108	6	290	292	K=	8,L=	8	9	137	136	9	160	178	5	89	92	10	105	97
10	149	152	4	110	132	7	269	283	10	96	81	10	229	225	11	103	123	9	93	103	K=	11,L=	2
11	329	334	5	183	196	8	193	169	16	124	113	11	318	324	16	99	104	10	200	193	0	125	109
12	106	114	K=	6,L=	7	9	84	95	17	110	121	12	105	82	17	111	130	11	152	122	2	89	64
13	130	115	3	115	137	10	237	235	K=	8,L=	7	18	101	113	K=	9,L=	2	12	154	171	9	128	134
15	239	237	5	105	90	11	130	126	5	192	195	K=	8,L=	1	1	158	142	13	113	113	K=	11,L=	3
17	135	130	K=	7,L=	10	12	297	309	7	118	89	0	153	150	5	104	110	15	88	93	1	95	88
18	329	343	9	103	86	13	104	105	9	109	93	1	224	229	6	100	116	16	110	91	6	219	258
21	210	219	19	91	75	14	156	125	11	190	194	5	141	169	7	197	212	19	107	68	K=	11,L=	4
22	140	154	K=	7,L=	9	15	164	172	12	116	126	7	139	104	9	306	289	20	89	58	4	87	56
K=	6,L=	2	5	118	73	17	113	115	15	173	163	8	159	161	10	101	125	K=	10,L=	2	K=	12,L=	5
1	169	181	12	201	210	19	92	111	16	169	172	9	119	160	14	179	187	2	88	23	10	97	62
2	392	388	15	162	173	21	138	128	18	113	113	11	106	95	16	103	111	4	204	219	K=	12,L=	4
3	245	235	27	116	77	K=	7,L=	1	K=	8,L=	6	12	206	194	17	101	81	5	113	124	1	91	96
5	419	424	K=	7,L=	8	1	149	143	1	145	170	13	148	141	K=	9,L=	1	8	275	261	4	116	65
6	290	271	5	143	143	2	195	186	3	115	96	16	112	103	1	143	165	9	194	188	7	141	154
7	503	503	8	144	154	4	157	155	6	138	126	K=	8,L=	2	3	81	41	10	87	133	12	95	70
8	188	174	9	120	31	7	206	206	7	194	193	0	123	133	4	133	169	12	101	108	K=	12,L=	3
9	118	113	10	139	139	8	77	71	8	133	111	1	196	214	7	263	250	14	226	231	8	149	113
10	115	110	12	143	153	9	118	115	9	263	255	2	347	363	8	143	135	K=	10,L=	1	K=	12,L=	2
14	250	237	14	151	145	11	512	509	10	127	120	3	200	178	9	203	174	5	85	94	2	128	129
15	88	82	22	120	129	13	119	97	11	103	81	7	96	87	10	111	105	7	267	270	9	148	150
16	237	231	27	149	138	14	278	270	12	303	312	8	143	142	12	226	224	10	88	126	11	93	111
19	119	121	K=	7,L=	7	15	189	180	13	184	190	9	108	105	13	111	108	11	86	55	K=	12,L=	1
21	158	150	6	160	169	16	242	233	14	106	91	12	95	76	14	132	119	17	101	36	1	108	68
K=	6,L=	1	8	275	283	17	201	221	20	108	98	13	112	110	15	122	99	18	92	50	3	96	42
1	473	474	9	208	192	19	88	49	24	117	122	K=	8,L=	3	K=	9,L=	0	K=	10,L=	0	4	97	93
2	428	422	11	160	153	23	101	72	26	126	121	0	236	251	1	154	162	0	129	173	8	140	122
3	187	155	15	198	198	K=	7,L=	0	K=	8,L=	5	1	87	90	2	103	104	1	297	289	11	131	97
4	169	181	16	106	129	1	150	155	1	104	81	2	230	237	3	172	201	2	156	150	K=	12,L=	0
5	509	486	18	150	166	2	437	434	2	164	177	4	110	91	6	125	151	3	87	104	7	153	134
6	84	97	19	101	70	3	235	226	3	107	80	5	113	122	7	178	168	5	143	139	9	107	35
7	239	244	21	109	139	4	210	209	5	85	56	8	101	109	8	102	125	7	93	137	K=	12,L=	1
8	70	43	22	114	96	5	164	167	7	128	125	9	127	135	9	176	192	8	106	153	0	84	56
10	257	250	24	114	60	6	153	148	12	256	271	10	92	95	10	164	155	12	102	113	4	120	99
11	155	142	27	115	114	8	215	234	14	373	390	11	121	118	11	142	145	13	127	123	K=	12,L=	2
13	222	228	K=	7,L=	6	11	297	293	17	188	184	K=	8,L=	4	12	164	142	16	93	47	0	166	180
14	363	356	1	156	141	12	195	205	19	90	60	0	128	113	16	96	73	K=	10,L=	1	1	91	66
18	272	276	4	155	154	15	88	105	K=	8,L=	4	1	89	54	K=	9,L=	1	2	232	237	3	112	94
19	126	115	5	280	264	16	118	95	1	98	35	2	127	149	2	156	144	4	231	234	K=	12,L=	3
21	131	155	6	315	330	17	103	81	2	77	24	4	110	122	3	228	214	6	120	137	0	101	68
22	92	77	9	176	179	18	92	102	3	192	184	10	119	116	4	228	230	7	102	110	K=	13,L=	1
K=	6,L=	0	10	322	338	20	106	115	5	134	141	K=	8,L=	5	5	99	111	11	117	114	4	94	59
0	360	355	11	115	111	K=	7,L=	1	7	235	253	0	109	70	6	79	56	K=	10,L=	2	K=	13,L=	0
1	399	383	14	267	260	0	211	217	8	362	357	1	172	170	8	156	151	0	114	112	1	97	48
2	82	65	15	89	91																		

Table 3. Interatomic distances, bond angles and dihedral angles with estimated standard deviations.

DISTANCE	(Å)	DISTANCE	(Å)	DISTANCE	(Å)
O1 - CC1	1.223(6)	O2 - CC2	1.214(6)	O3 - CC3	1.219(5)
O4 - CC4	1.225(5)	O5 - CC5	1.218(6)	O6 - CC6	1.223(6)
O7 - CC7	1.224(6)	CC1 - N7	1.352(6)	CC2 - N1	1.333(6)
CC3 - N2	1.335(6)	CC4 - N3	1.336(6)	CC5 - N4	1.342(6)
CC6 - N5	1.357(6)	CC7 - N6	1.347(6)	CC1 - C1	1.531(7)
CC2 - C2	1.525(8)	CC3 - C3	1.525(8)	CC4 - C4	1.524(7)
CC5 - C5	1.530(7)	CC6 - C6	1.523(7)	CC7 - C7	1.531(7)
C1 - N1	1.449(7)	C2 - N2	1.436(6)	C3 - N3	1.438(7)
C4 - N4	1.461(6)	C5 - N5	1.467(6)	C6 - N6	1.450(6)
C7 - N7	1.446(6)	N1 - CM1	1.438(7)	N2 - CM2	1.454(7)
N3 - CM3	1.454(7)	N4 - CM4	1.438(7)	N5 - CM5	1.455(7)
N6 - CM6	1.440(7)	N7 - CM7	1.444(8)	Ow - O5	2.833(6)
Ow - O4	2.864(6)				

ANGLE	(°)	ANGLE	(°)
O1 - CC1 - C1	121.2(5)	O2 - CC2 - C2	120.1(5)
O3 - CC3 - C3	119.9(5)	O4 - CC4 - C4	120.8(5)
O5 - CC5 - C5	120.3(5)	O6 - CC6 - C6	120.1(5)
O7 - CC7 - C7	121.1(5)	O1 - CC1 - N7	122.3(5)
O2 - CC2 - N1	123.7(6)	O3 - CC3 - N2	123.0(5)
O4 - CC4 - N3	122.8(5)	O5 - CC5 - N4	123.6(5)
O6 - CC6 - N5	123.0(5)	O7 - CC7 - N6	121.5(5)
CM1 - N1 - C1	117.4(5)	CM2 - N2 - C2	115.3(5)
CM3 - N3 - C3	115.6(5)	CM4 - N4 - C4	117.8(5)
CM5 - N5 - C5	117.1(5)	CM6 - N6 - C6	116.8(5)
CM7 - N7 - C7	116.8(5)	CM1 - N1 - CC2	118.4(5)
CM2 - N2 - CC3	118.3(5)	CM3 - N3 - CC4	123.5(5)
CM4 - N4 - CC5	123.7(5)	CM5 - N5 - CC6	121.1(5)
CM6 - N6 - CC7	118.9(5)	CM7 - N7 - CC1	118.8(5)
N7 - CC1 - C1	116.4(5)	N1 - CC2 - C2	116.2(5)
N2 - CC3 - C3	117.1(5)	N3 - CC4 - C4	117.1(5)
N4 - CC5 - C5	116.8(5)	N5 - CC6 - C6	116.9(5)
N6 - CC7 - C7	117.4(5)	C1 - N1 - CC2	123.2(5)
C2 - N2 - CC3	125.3(5)	C3 - N3 - CC4	120.4(5)
C4 - N4 - CC5	118.2(4)	C5 - N5 - CC6	116.6(5)
C6 - N6 - CC7	121.7(4)	C7 - N7 - CC1	124.5(5)
CC1 - C1 - N1	112.1(5)	CC2 - C2 - N2	110.1(4)
CC3 - C3 - N3	114.1(5)	CC4 - C4 - N4	111.8(5)
CC5 - C5 - N5	109.4(4)	CC6 - C6 - N6	111.7(5)
CC7 - C7 - N7	111.2(5)	Ow - O5 - CC5	143.6(4)
Ow - O4 - CC4	139.2(4)	O5 - Ow - O4	103.8(2)

DIHEDRAL ANGLE	(°)	DIHEDRAL ANGLE	(°)
CC1 - C1 - N1 - CC2	86.7(6)	C1 - N1 - CC2 - C2	1.4(8)
N1 - CC2 - C2 - N2	179.6(5)	CC2 - C2 - N2 - CC3	-82.8(7)
C2 - N2 - CC3 - C3	-9.5(8)	N2 - CC3 - C3 - N3	-68.7(7)
CC3 - C3 - N3 - CC4	134.4(5)	C3 - N3 - CC4 - C4	175.9(5)
N3 - CC4 - C4 - N4	-175.7(4)	CC4 - C4 - N4 - CC5	-87.4(6)
C4 - N4 - CC5 - C5	-179.8(5)	N4 - CC5 - C5 - N5	168.7(5)
CC5 - N4 - N5 - CC6	63.8(7)	C5 - N5 - CC6 - C6	-161.2(4)
N5 - CC6 - C6 - N6	177.1(4)	CC6 - C6 - N6 - CC7	-72.1(6)
C6 - N6 - CC7 - C7	-13.2(7)	N6 - CC7 - C7 - N7	-163.5(5)
CC7 - C7 - N7 - CC1	-92.4(6)	C7 - N7 - CC1 - C1	-8.6(7)
N7 - CC1 - C1 - N1	179.9(5)		

Table 4.

Distance (Å)	(I)	(II)	(III)	Cycloheptasarcosyl
CC—C	1.527	1.530	1.525	1.534
CC—N	1.344	1.345	1.346	1.343
CC—O	1.228	1.232	1.224	1.221
C—N	1.456	1.453	1.454	1.449
CM—N	1.483	1.487	1.466	1.446

Table 5.

Angle (°)	(I)	(II)	(III)	Cycloheptasarcosyl
(CM—N—CC) *cis*	118.7	118.6	117.9	118.6
(CM—N—CC) *trans*	123.9	123.5	123.8	122.8
(C—N—CC) *cis*	123.8	122.8	123.5	123.7
(C—N—CC) *trans*	117.2	117.5	116.1	118.4

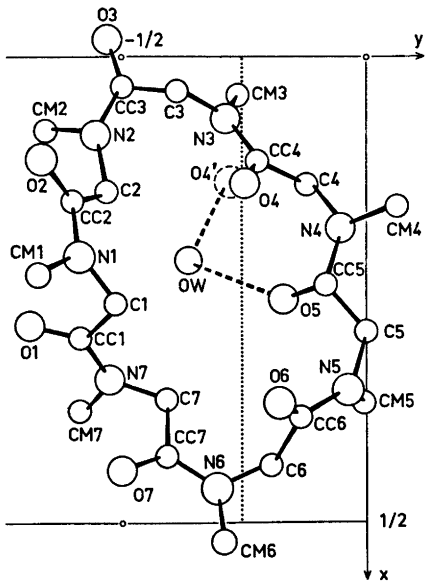


Fig. 1.

squares refinement cycle. Fig 1 shows the molecule viewed along [001].

By averaging bond distances of Table 3, and comparing with the results of the corresponding pentameric [6] (I) and octameric [5] (II) compounds, and with cycloalanyltetrasarcosyl [11] (III), no significant differences are observed (Table 4). The somewhat longer CM—N distances of (I) and (II) are possibly connected with the fact that for these compounds, methyl hydrogens were not included in the calculations. The geometry of the *cis* and *trans* *N*-methyl amide groups, respectively, is also roughly the same (Table 5).

Fig. 1 shows that the ring conformation is *cis,cis,cis,cis,trans,trans,trans*, quite unexpectedly a widely different conformation from that of cyclooctasarcosyl [5] (*cis,cis,trans,trans,cis,cis,trans,trans*).

The water molecules link glide plane equivalent molecules to endless chains along [001]. The two O···O distances are approximately equal (OW···O_5=2.833 Å; OW···O_4'=2.864 Å) and the angle O_5···OW···$O^{3'}$ is 103.8°.

Since the shortest CC···N distance across the ring is longer than 3.5 Å, no direct transannular contact can be held responsible for the rigidity of this 21-membered ring. As in earlier findings,[4–6,11] the explanation must be sought in the intrinsic conformation of the peptide chain itself.[2]

Apart from the hydrogen bonds, there are no short inter-molecular contacts.

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